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COMPATIBILITY AND DECONTAMINATION OF HIGH-DENSITY POLYETHYLENE EXPOSED TO SULFUR MUSTARD

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The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorizing documents.

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14. ABSTRACT:

The Environmental Monitoring Laboratories, in cooperation with the Chemical Operations Branch of the U.S. Army Edgewood Chemical Biological Center (ECBC) Directorate of Program Integration (DPI), conducted a multi-week study to determine the compatability of high-density polyethylene (HDPE) with liquid mustard (HD) material and decontamination of HDPE when exposed to HD. In Part I of the experiment, we tested the compatability of HD with HDPE to determine the amount of HD adhered or adsorbed onto the material and to observe any physical degradation of the material when exposed to HD over time. In Part II of the experiment, we evaluated the ability of HDPE to be decontaminated after it had been in contact with HD for short and extended periods of time. Both parts of this experiment will be used to determine the feasibility of utilizing HDPE in the construction of universal munition storage containers for leaking and nonleaking HD munitions until their eventual neutralization in the Explosive Destruction System.

15. SUBJECT TERMS

Explosive Destruction System (EDS)	Mustard (HD)
High density polyethylene (HDPE)	universal munition storage containers (UMSC)

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PREFACE

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CONTENTS

FIGURES

TABLES

COMPATIBILITY AND DECONTAMINATION OF HIGH-DENSITY POLYETHYLENE EXPOSED TO SULFUR MUSTARD

1. INTRODUCTION

The Explosive Destruction System ([EDS] shown in Figure 1), developed by the U.S. Army Chemical Materials Activity, is utilized by the U.S. Army Edgewood Chemical Biological Center to destroy chemical warfare material in an environmentally safe manner and with no adverse affects to its operators. The system uses cutting charges to explosively access chemical munitions prior to their chemical neutralization. One of the challenges in EDS operations is the transport and insertion of munitions that have developed leaks. These leaking munitions pose hazards to workers and the environment. Therefore, establishing a method to handle the leaking munitions safely is extremely important. To combat this hazard, the construction of a universal munition storage container (UMSC) was proposed.

For this project, high-density polyethylene (HDPE) was chosen for the construction of the UMSC. Leaking and nonleaking mustard (HD) munitions were stored in the UMSC until destruction in the EDS. The UMSC containing the munitions was placed directly in the EDS and was destroyed along with the munition, thereby eliminating direct handling of the munitions, leaking or otherwise. This experiment evaluates the compatability of HD and HDPE and the ability to decontaminate the material after a simulated bench-scale EDS operation.

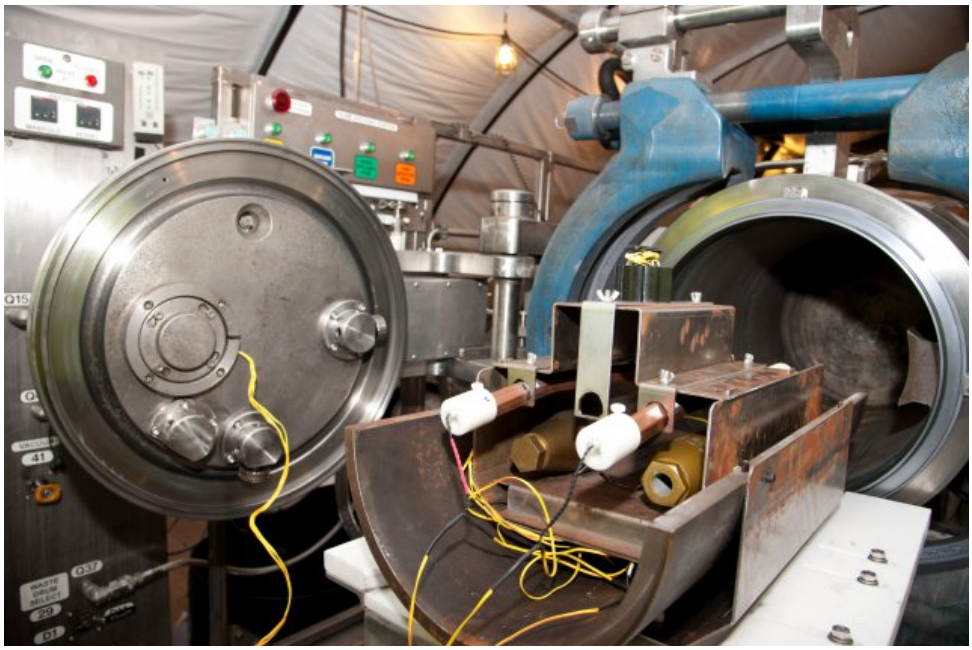

Figure 1. Two munitions being placed in the EDS.

2. OBJECTIVES

2.1 Task 1–HDPE Compatibility with HD

The primary purposes of Task 1 were to visually observe the compatibility of HD with HDPE and to measure weight changes over time in the HDPE after it was soaked in HD. An additional goal was to determine whether the amount of HD adsorbed onto and absorbed into the coupon increased with time.

For this task, 32 random HDPE coupons, cut from smooth and jagged exploded pieces of UMSCs and approximately 1–2 g in mass, were placed in 40 mL volatile organic analysis (VOA) vials and weighed. Each coupon was spiked with 2 mL of HD (Figure 2). The samples were placed in storage at ambient temperature for time periods ranging from 1 to 12 weeks.

Once weekly, 2 sample coupons were removed from storage for observation. Each sample was photographed, and any observational changes to the HD and HDPE were recorded. The coupon was then removed from the HD, towel-dried, and weighed. Each coupon was placed in a new VOA vial and rinsed with hexane. The hexane rinsate was diluted and analyzed for HD.

2.2 Task 2–Decontamination of HPDE and HD by EDS Simulated Treatment

Task 2 evaluated the ability to decontaminate the HDPE that had come into contact with HD for short and extended time periods.

Approximately 40 HDPE coupons, cut from smooth and jagged exploded pieces of UMSCs and approximately 1–2 g in mass, were placed in VOA vials and weighed. Twenty coupons were spiked with 2 mL of HD and put aside for 30 min on the same day that they were treated.

The remaining 20 coupons were spiked with 2 mL of HD and stored at ambient temperature for 35–56 days, with 5 coupons treated during each operational week of the study. Twenty milliliters of monoethanolamine (MEA) was added to each coupon at 60 ^{o}C for 1 h. The MEA was decanted, followed by the addition of 20 mL of water at 60–95 ^{o}C for 1 h. The water was then decanted. This process closely mimicked the destruction process used in the EDS.

The drained MEA and water rinse were extracted and analyzed separately to determine the residual concentration of HD in each matrix. A treatment goal of 50 ppm (50,000 µg/L) in MEA for the EDS neutralent was established.

Following the water drain, the coupon was vapor-washed with nitrogen for 15 min, placed in a 10 × 10 in. plastic bag, sealed, and allowed to off-gas for 1 h. A 10 L vapor sample was collected using thermal desorption tubes and analyzed for HD. The coupon was placed inside a clean VOA vial and rinsed with hexane. The hexane rinsate was analyzed to

determine whether any residual HD could be recovered. Finally, the coupon was dried with laboratory towels and the final coupon weight was recorded.

3. EXPERIMENTAL PROCEDURES

3.1 Task 1–Compatibility of HD and HDPE

The following steps were used to determine the compatibility of HD and HDPE:

a. Two coupon vials were removed from storage and photographed (Figure 3).

b. Each coupon was observed for physical changes, and the coupon and HD. Were photographed. All observations were recorded in a logbook.

c. The HD was removed from the sample container using a pipette. The aspirated HD was placed directly into bleach decon.

d. The coupons were dried with laboratory towels (to prevent seepage, aluminum foil was placed under the towels).

e. The dried coupon was transferred to a tared 40 mL VOA vial, and the weight was recorded.

f. The coupon was rinsed with 40 mL of hexane.

g. The hexane was decanted into a clean 40 mL VOA vial and put aside.

h. The coupon was dried with laboratory towels and transferred to a tared 40 mL VOA vial, and the weight was recorded.

i. 0.5 mL of the hexane rinse from step g was added to a clean 40 mL VOA vial. An additional 40 mL of hexane was added (this diluted the sample to the point that it could be accurately quantitated within the instrument calibration range).

j. 1.9 mL of the diluted rinse and 100 µL of internal standard were transferred into a clean 16 mm centrifuge tube. The solution was vortexed, transferred into a 2 mL amber vial, and analyzed for HD in accordance with (IAW) the Environmental Monitoring Laboratory ([EML) Internal Operating Procedure (IOP) MT-60.[1]

[1] Dusick, B. *Analysis of Residual Sulfur Mustard (HD) and HD Breakdown Products in EDS Neutralent/Waste Including Monoethanolamine (MEA)*; IOP MT-60; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2014 (in-house IOP for the Directorate of Program Integration - Environmental Monitoring Laboratory, ECBC).

k. Assessed coupons were placed in storage for later disposition.

Figure 2. Task 1 coupons spiked with 2 mL of HD for storage.

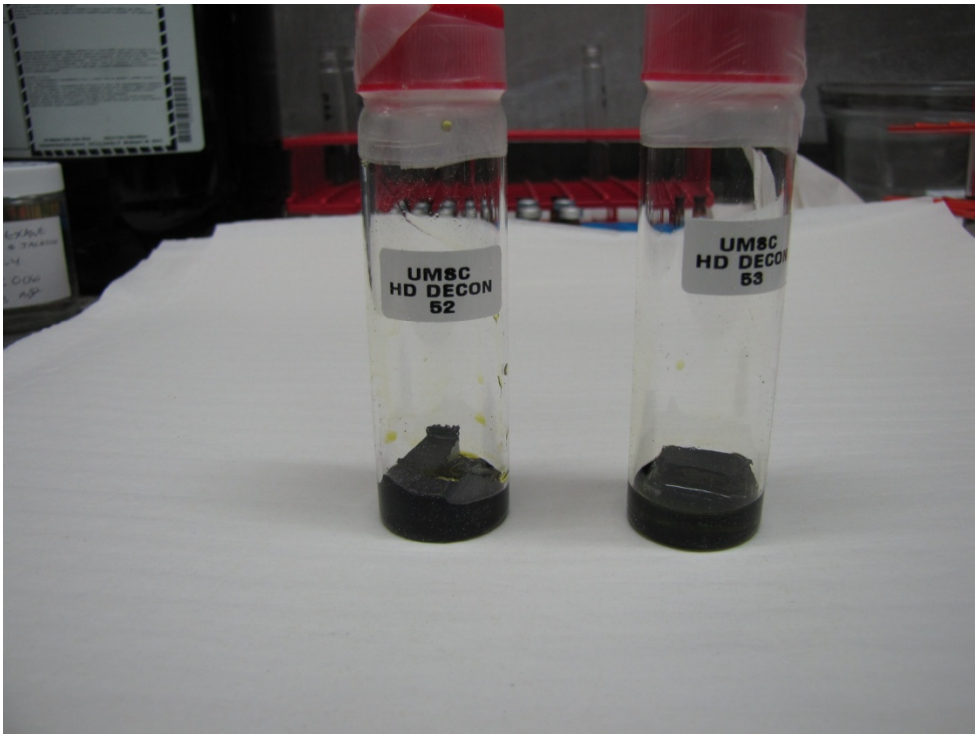

Figure 3. Task 1 coupons after 27 days of storage. The dark green color was caused by oxidation due to the presence of copper in the HDPE.

3.2 Task 2–Decontamination and Treatment of Coupons

For the initial testing, the coupons were allocated in the following manner:

- 5 coupons were treated at the bench-scale level each week of this study.
- Coupon numbers 11–25 and 78–82 were spiked on the same day as treatment.
- Coupon numbers 26–45 were spiked and placed in storage at room temperature for 35–56 days.

The following steps were used to decontaminate and treat the coupons:

a. A sand bath was heated to 60 ^{o}C. The bath was placed on top of a hot plate equipped with a stirring plate, and a thermometer was inserted in the sand (Figure 6).

b. The MEA and water were preheated on a hot plate with a stir bar.

c. For same-day samples, 2 mL of HD was added to each coupon and put aside for 30 min. (Holding-time samples only need to be removed from storage.) The coupons were photographed before the MEA was added (Figure 4).

d. 20 mL of MEA at ~60 ^{o}C and a small stir bar (12.7 mm length × 3.2 mm diameter) were added to each sample container (Figure 5).

e. The sample containers were placed in the sand bath and insulated with aluminum foil to retain heat (Figure 6).

f. The stir plate was turned on and set to 2200 rpm. (This stirring was done to simulate the cavitation caused by the rotation of the EDS vessel. Initial testing showed that without stirring, the reaction of the MEA and HD did not meet the 50 ppm target.)

g. A thermometer was inserted in the sand and the temperature was recorded every 5 min. The hot plate was adjusted, as necessary, to keep the temperature at ~60 ^{o}C.

h. After 1 h, MEA was decanted into another 40 mL VOA vial.

i. The MEA neutralent was extracted and analyzed for HD IAW EML IOP MT-60.[1]

j. 20 mL of water was added at ~95 ^{o}C to each sample container.

k. The sample containers were replaced in the sand bath at 60 °C. The temperature was recorded every 5 min.

l. After 1 h, water was decanted into a 40 mL VOA vial.

m. The water sample extracted and analyzed for HD IAW procedures found in EML IOP MT-60.[1]

n. The coupon was vapor washed in a sample bottle with nitrogen for 15 min and placed in a 10 × 10 in. plastic bag for 1 h.

o. A 10 L sample was collected using a thermal desorption tube and analyzed for HD IAW EML IOP MT-13 (Figure 7).[2]

p. The coupon was placed in a clean 40 mL VOA vial, and 1.9 mL of hexane, and 100 µL of internal standard were added. The hexane rinse was transferred to a 2 mL amber vial and analyzed for HD IAW EML IOP MT-60.[1]

q. The coupon was dried with laboratory towels and the final weight was recorded. The final coupons were photographed.

[2] Directorate of Program Integration. *Analysis of Chemical Warfare Agents and Degredation Products on DAAMS Tubes Using a Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)*; IOP MT-13; rev. 1; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2010 (in-house IOP for the Directorate of Program Integration - Environmental Monitoring Laboratory, ECBC).

Figure 4. Task 2 coupons and a metal blank spiked with HD before treatment. A metal blank was used in two experiments to verify that the HDPE was not interfering with the reaction between HD and MEA.

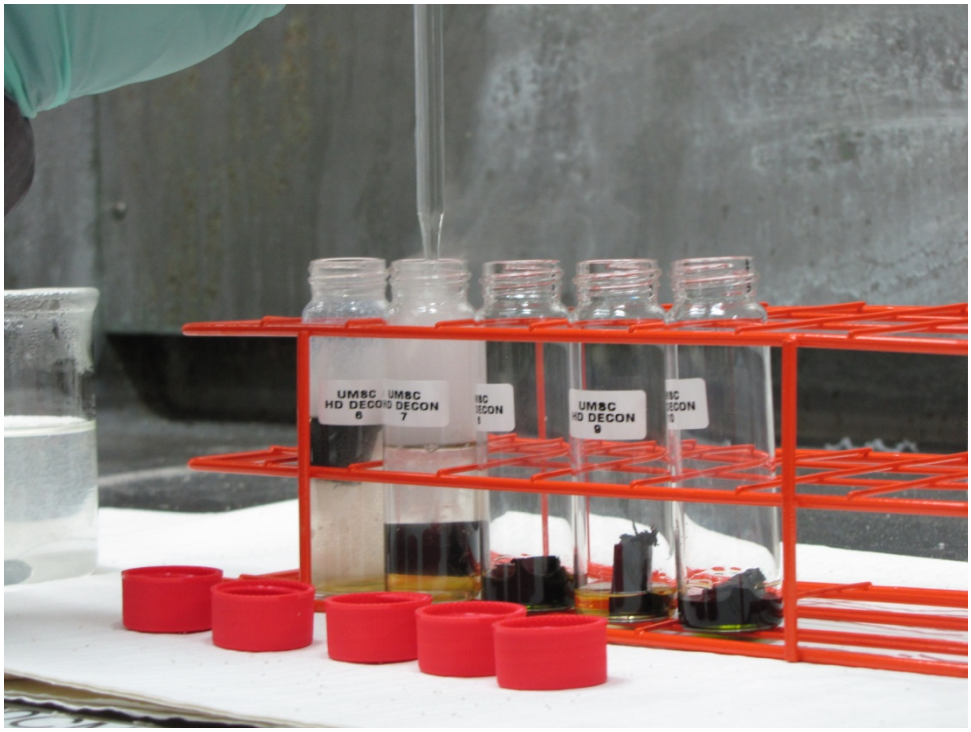

Figure 5. Addition of MEA.

Figure 6. Reaction sand bath. The bowl rested on top of the hot plate. Each VOA vial had a small stir bar inside. Foil was wrapped around the bowl and placed on top for insulation and temperature control. A thermometer was inserted in the sand bath for temperature monitoring.

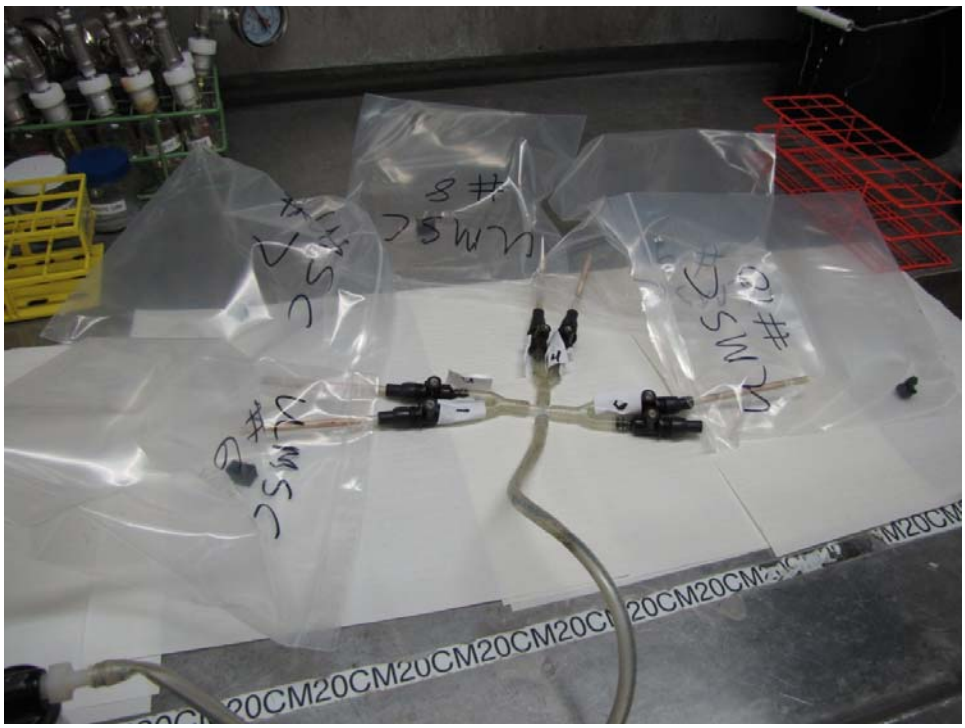

Figure 7. Depot area air monitoring system (DAAMS) tube sampling of coupons. A 10 L air sample was collected to mimic the Tedlar bag air sampling of the EDS, which is used to verify that it is safe to open the EDS door. Samples were collected at a flow rate of 500 mL/min for 20 min.

4. RESULTS

The results of the decontamination and compatibility tests can be found in Tables 1–3. The data for coupon numbers 1–10 (HD treatment goals were not met) were used to fine-tune the decontamination procedure conditions. It was determined that a sand bath that fully encapsulated the 40 mL VOA vial and the addition of a stir bar (used to simulate the cavitation caused by a rotating EDS vessel) were necessary to more accurately simulate the EDS destruction operation. The results from those failed trials are not included in the data tables.

Table 1. HD and HDPE Compatibility Data

Coupon Name and Number		Start Weight (g)	Holding Time (days)	Weight after Removing HD (g)	[HD] in Hexane Rinse (µg/L)	HD Amount from Coupon (g)	End Weight of Coupon (g)
UMSC	**46**	1.36	5	1.36	259,200	0.010	1.35
	47	1.22	5	1.23	160,810	0.0064	1.23
	48	1.41	13	1.42	179,560	0.0072	1.41
	49	1.23	13	1.24	81,170	0.0032	1.24
	50	1.12	20	1.13	169,250	0.0068	1.12
	51*	1.48	20	1.49	123,290	0.0049	1.49
	52*	1.59	27	1.62	378,340	0.015	1.60
	53*	1.35	27	1.37	301,750	0.012	1.36
	54	1.14	35	1.15	121,500	0.0049	1.15
	55	1.33	35	1.36	263,890	0.011	1.35
	56	1.28	41	1.30	78,020	0.0031	1.30
	57	1.74	41	1.71	102,060	0.0041	1.71
	58*	1.74	48	1.81	381,380	0.015	1.81
	59*	1.52	48	1.53	500,410	0.020	1.53
	60*	1.66	56	1.79	254,430	0.010	1.78
	61	1.24	56	1.27	130,110	0.0052	1.27
	62*	2.10	60	2.11	104,440	0.0042	2.11
	63*	1.47	60	1.50	442,530	0.018	1.49
	64*	1.62	69	1.64	199,810	0.0080	1.64
	65*	2.02	69	2.05	276,170	0.011	2.05
	66*	1.01	76	1.03	235,050	0.0094	1.02
	67*	1.06	76	1.08	105,510	0.0042	1.07
	68*	1.11	83	1.13	362,960	0.015	1.13
	69*	1.15	83	1.18	337,180	0.014	1.18

*Samples showed partial or full oxidation, as indicated by a color change from orange to green.

Table 2. Decontamination Data–Same Day Treatment

Coupon Name and Number		Start Weight (g)	MEA Reaction Time (min)	[HD] in MEA (µg/L)	[HD] in Water (µg/L)	Vapor Screen (mg/m^3)	[HD] in Hexane Rinse (µg/L)	End Weight (g)
UMSC	11	1.61	140	170 J	150 J	0.003 E	230 J	1.62
	12	1.46	140	170 J	150 J	0.005 E	380 J	1.47
	13	1.53	140	180 J	150 J	0.0009	250 J	1.53
	14	1.58	140	170 J	150 J	0.0008	230 J	1.58
	15	1.47	140	200 J	150 J	0.001	300 J	1.47
	HD blank	NA	90	150 J	NA	NA	NA	NA
	HD blank*	NA	90	150 J	NA	NA	NA	NA
	16	1.86	92	170 J	<500	<4.8E-4	320 J	1.87
	17	1.46	92	160 J	<500	<4.8E-4	380 J	1.47
	18	1.63	92	160 J	<500	0.0006	420 J	1.64
	19	2.08	92	170 J	<500	0.0007	360 J	2.09
	20	1.13	92	190 J	<500	0.0009	510	1.14
	HD blank*	NA	60	<500	NA	NA	NA	NA
	21	1.72	60	180 J	<500	0.0013	280 J	1.72
	22	1.14	60	160 J	<500	0.0009	310 J	1.15
	23	1.40	60	180 J	<500	0.004 E	500	1.40
	24	1.31	60	170 J	<500	0.003 E	370 J	1.32
	25	1.25	60	160 J	<500	0.001	240 J	1.25
	Metal blank **	5.29	60	<500	<500	<4.8E-4	<500	5.29
	78	1.23	60	490 J	<500	0.0008	920L	1.25
	79	1.30	60	250 J	<500	0.001	770	1.31
	80	1.20	60	340 J	<500	0.0006	410 J	1.20
	81	1.85	60	340 J	<500	0.003 E	1490	1.86
	82	1.82	60	450 J	<500	0.004 E	12,920 D	1.83
UMSC **	Metal blank **	5.39	60	<500	<500	<4.8E-4	<500	5.39

Qualifiers: "J", Analyte detected in sample greater than the method detection limit (MDL), but less than the laboratory limit of quantitation (LOQ).
"E", Analyte detection exceeds highest calibration point resulting in an estimated value.
"D", Sample required a dilution to accurately quantitate the result within the calibration curve.
* HD blanks were vials with no HDPE coupons that contained 2 mL of HD to which 20 mL of MEA was added for reaction product analysis only. The process was stopped before step j in Section 3.2.
**The metal blanks consisted of a small piece of metal that was added to a 40 mL VOA vial and taken through the entire decontamination procedure.
***For coupons 11–20, the water temperature was ~65 ^{o}C when added. In all subsequent trials, the water temperature was ~ 95 ^{o}C.
NA, not applicable.

Table 3. Decontamination Data–Pretreated Coupons

Coupon Name and Number		Starting Weight (g)	Holding Time (days)	[HD] in MEA (µg/L)	[HD] in Water (µg/L)	Vapor Screen (mg/m^3)	[HD] in Hexane Rinse (µg/L)	Ending Weight (g)
UMSC	**26***	1.91	35	4700	<500	0.008 E	3840	1.92
	27	1.22	35	1890	<500	0.008 E	2600	1.23
	28	1.29	35	14,700 D	<500	0.006 E	3580	1.29
	29	1.46	35	48,170 D	<500	0.008 E	2880	1.47
	30	1.51	35	32,960 D	<500	0.004 E	5650	1.52
	31*	1.10	41	5560	<500	0.0012	3630	1.11
	32*	1.18	41	6200	210	0.0010	1880	1.19
	33*	0.86	41	14,330 D	<500	0.0006	10,720 D	0.87
	34*	1.44	41	19,560 D	<500	0.0014	25,850 D	1.45
	35	1.15	41	14,870 D	210 J	0.0001	1980	1.16
	36	1.39	48	7770	215 J	0.0017	6480	1.43
	37	1.34	48	7910	<500	0.0007	2330	1.35
	38*	1.40	48	11,240 D	<500	0.0034 E	3670	1.40
	39	1.30	48	14,040 D	<500	0.0016	3060	1.31
	40	1.17	48	16,760 D	<500	0.0018	5790	1.18
	41*	1.36	56	9140	210 J	0.0074 E	652,000 D	1.39
	42	1.74	56	1390	<500	0.0083 E	6520	1.75
	43	1.26	56	613	<500	0.015 E	5670	1.28
	44	1.23	56	2680	<500	0.009 E	3750	1.25
	45*	1.24	56	4710	<500	0.0063 E	3710	1.26

Qualifiers: "J", Analyte detected in sample greater than the MDL, but less than the laboratory LOQ.
"E", Analyte detection exceeded highest calibration point, resulting in an estimated value.
"D", Sample required a dilution to accurately quantitate the result within the calibration curve.
*Samples showed partial or full oxidation, as indicated by a color change from orange to green.

5. CONCLUSIONS

5.1 Task 1–HD and HDPE Compatibility

1. HD either adhered to and/or was absorbed by the HDPE coupons. To some degree, HD was recovered by washing with hexane.

2. HD did not cause any observable visual degradation in the HDPE.

3. The amount of HD absorbed/adsorbed onto the coupons did not increase significantly over time (Figure 8). Most likely, the data fluctuations can be attributed to the drying step of the procedure.

4. The HDPE coupons did not show significant weight increase after the HD drying step or after the hexane rinse step.

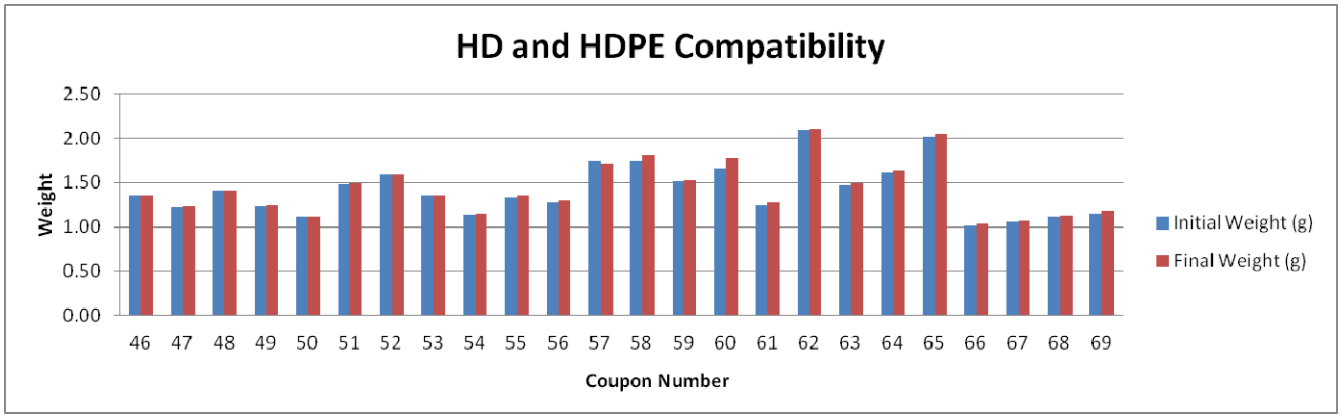

Figure 8. Comparison of initial and final coupon weights.

5.2 Task 2–Decontamination

5.2.1 Same-Day Treatment

Data analyses for same-day decontamination yielded the following results:

1. The HDPE did not interfere with the neutralization of agent in the EDS bulk reaction liquid, and the HD concentration of <50 ppm (50,000 µg/L) treatment goal was achieved for the neutralent.

2. There was little to no change in the before and after coupon weights. Comparison with the metal blanks showed that residual HD adhered to the HDPE coupons throughout the decontamination procedure and could still be detected at low levels after MEA decontamination and water rinsing. In almost all cases, HD was detected on the DAAMS tubes sampling the Tedlar bag vapor. This suggests that clearing the inside of the vessel to open the door may be an issue in EDS operations.

5.2.2 Pretreated Coupons

Data analyses of pretreated coupons yielded the following results:

1. The HDPE did not interfere with the established EDS reaction conditions and the HD concentration of <50 ppm (50,000 µg/L) treatment was achieved.

2. There was little to no change in the beginning and ending coupon weights.

3. In all cases, HD was detected on the DAAMS tubes sampling the Tedlar bag.

4. The amounts of HD detected in the MEA and hexane rinses were significantly higher in the pretreated coupons than in the same-day treated coupons, showing that the HD adhered more to the HDPE over time. However, the increased HD-HDPE contact time did not increase the amount of absorbed HD.

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ACRONYMS AND ABBREVIATIONS

ECBC	U.S. Army Edgewood Chemical Biological Center
DAAMS	depot area air monitoring system
EDS	Explosive Destruction System
EML	Environmental Monitoring Laboratory
HD	distilled mustard agent
HDPE	High-density polyethylene
IAW	in accordance with
IOP	Internal Operating Procedure
LOQ	limit of quantitation
MDL	method detection limit
MEA	monoethanolamine
VOA	volatile organic analysis
UMSC	universal munition storage container

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APPENDIX

POSTEXPLOSIVE DESTRUCTION SYSTEM TESTING

In addition to the bench-top experiment (Task 2), a full-scale Explosive Destruction System (EDS) destruction test of HD (distilled mustard agent) containers encapsulated in high-density polyethylene (HDPE) universal munition storage containers (UMSCs) was conducted. The Directorate of Program Integration - Environmental Monitoring Laboratory (ECBC) team was able to obtain 10 HDPE coupons from those containers. Five coupons were cut from the jagged HDPE pieces located directly below the linear charges, and five were cut from the smooth HDPE pieces located away from the charges.

After receipt of the coupons, a vapor screen by depot area air monitoring system tubes and a hexane rinse were performed, mimicking the Task 2 procedure steps n–p in Section 3.2. The results are shown in the Table.

Table. Post EDS/UMSC Test

Sample Name and Number		**Vapor Screen (mg/m^3)**	**[HD] in Hexane Rinse (µg/L)**	**Coupon Type**
UMSC	**101**	<4.76 E-4	240 J	Smooth
	102	<4.74 E-4	230 J	Smooth
	103	<4.77 E-4	230 J	Smooth
	104	<4.75 E-4	230 J	Smooth
	105	<4.76 E-4	230 J	Smooth
	106	6.85 E-4	800	Jagged
	107	8.31 E-4	640	Jagged
	108	6.46 E-4	620	Jagged
	109	7.92 E-4	620	Jagged
	110	8.05 E-4	520	Jagged


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